

A Research Report.

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**Application of Sputter-Deposited  
Lamellar Composite Technology  
to the Development of High  
Temperature Turbine Blade  
Materials and Airfoil Fabrication**

**Annual Technical Report: October 1976**

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**R. Busch and J. W. Patten**

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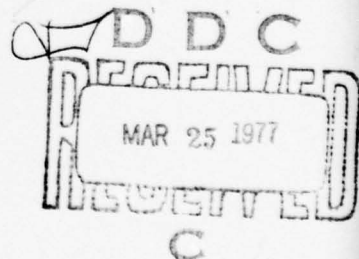
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APPLICATION OF SPUTTER-DEPOSITED LAMELLAR COMPOSITE  
TECHNOLOGY TO THE DEVELOPMENT OF HIGH  
TEMPERATURE TURBINE BLADE MATERIALS AND AIRFOIL FABRICATION

R. Busch and JW Patten

ABSTRACT

An experimental study of sputter-deposited lamellar composite materials and their potential application to gas turbine blading is described. The composite material systems investigated involved a refractory metal and an intermetallic compound based on the refractory metal and a strong oxide forming element. The compound layers were expected to form protective scales and thus protect as well as strengthen the refractory metal. In three systems (W-W<sub>2</sub>Zr, Mo-Mo<sub>2</sub>Zr, and Nb-Nb<sub>3</sub>Al) the compound did not exhibit protective behavior, in the fourth (Nb-NbBe<sub>12</sub>) the thermal stability of the composite was inadequate.

Part of the study was devoted to the demonstration of a soluble mandrel method for fabricating airfoil shapes bonded to cast platform-and-root sections by sputter deposition. Bonded airfoils of ~ 15 mil wall thickness were produced, but several difficulties encountered prevented a satisfactory demonstration of the technique.

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APPLICATION OF SPUTTER-DEPOSITED LAMELLAR COMPOSITE  
TECHNOLOGY TO THE DEVELOPMENT OF HIGH-  
TEMPERATURE TURBINE BLADE MATERIALS AND AIRFOIL FABRICATION

INTRODUCTION

The general objective of this program was to determine the feasibility of forming lamellar composite materials with useful engineering properties via sputter deposition. In the first phase of the program,<sup>(1,2)</sup> the mechanical properties of two types of lamellar composite were investigated. In the first type, the properties resulted from the dimensions of the lamellae which were  $< 100 \text{ \AA}$  thick. The second type more nearly resembled conventional composites, in that the properties were due to reinforcing the major constituent with a second constituent of higher elastic modulus. Both types of composites exhibited high strengths and limited plasticity, and their layered structures were stable at temperatures on the order of  $0.7 T_m$ .

These results, together with the high priority accorded gas turbine blading by the armed services, strongly suggested the direction of the second phase of the program, which was intended to demonstrate the application of lamellar composites to current DOD problems. The proposed effort consisted of two parts -- development of a lamellar composite material system for high temperature service, and demonstration of turbine blade fabrication via the sputter deposition of a lamellar composite.



## MATERIAL DEVELOPMENT

### SELECTION OF MATERIAL SYSTEMS

The material development work was based on the concept of a refractory metal strengthened and protected from oxidation by an intermetallic compound of the refractory metal with a strong oxide former. This concept is suitable only for a composite of the present configuration, where continuous layers are deposited alternately, and not to composites of the matrix-plus dispersed reinforcement type.

Alloy systems were selected according to the following criteria:

- 1) Melting points of the refractory metal and the selected compound.
- 2) Thermodynamic stability of the composite, e.g. the compound should be adjacent to the refractory metal in the phase diagram and there should be small mutual solubility.
- 3) The oxide forming element should yield an oxide whose thermal expansivity is similar to that of the refractory metal and intermetallic compound.
- 4) The density should be the lowest available consistent with satisfying the above criteria.

Examination of available binary phase diagrams<sup>(3-5)</sup> yielded three systems which potentially met these criteria. They were W-W<sub>2</sub>Zr, Mo-Mo<sub>2</sub>Zr and Nb-Nb<sub>3</sub>Al, in order of descending melting points and relative stability (based on mutual

solubility). Deposits of the intermetallic compounds were tested for oxidation behavior in still air at 1000-1200°C. The deposition conditions and results are listed in Table I.

#### SPUTTER DEPOSITION

W-Zr and Mo-Zr deposits were formed from double "dee" targets of the elements arranged as shown in Figure 1. This arrangement produced a deposit with a composition gradient across the diameter normal to the target division line. The desired composition was located by x-ray fluorescence. Samples were cut from this area of the deposit for oxidation testing. The presence of the intermetallic compound was verified by x-ray diffraction.

#### EVALUATION OF OXIDATION BEHAVIOR

##### Mo<sub>2</sub>Zr

The Mo<sub>2</sub>Zr specimens exhibited large weight losses upon their initial air exposure (20 minutes at 1200°C) and no further change on subsequent exposures. This behavior was observed both in as-deposited specimens and a group that had been vacuum annealed to ensure completion of the compound forming reaction prior to air exposure. The weight loss roughly corresponded to loss of the molybdenum less a small increase due to oxidation of the zirconium.

In a separate experiment, a Mo<sub>2</sub>Zr specimen was placed in a quartz tube which was partially evacuated to give a pressure of one atmosphere of air at 1200°C. The tube was sealed and placed in a furnace so that the end containing

TABLE I. Deposition Conditions and Results for Refractory Intermetallics

Material & Exp. No.	Target Amp-Hrs	Grams Deposited	Deposit Thickness (mils)	Results
ZrW-1	1.25	2.9	1.1	Shut down by shield peeling
ZrW-2	< 0.8	--	--	Deposit debonded
ZrMo-1	2.0	4.5	2.9	Shut down by shield peeling
-2	4.1	9.9	5.4	Shut down by target short circuit, shields peeled when system opened
-3	~ 0	--	--	Short circuit, not due to peeling
-4	5.7	13.5	7.6	Shut down by peeling of target shield
-5	2.1	.8	3.1	Cumulative thickness 19 mils
NbBe-1	4.3	4.5	6.1	Normal deposition, off desired composition
-2	5.0	5.2	9.0	Normal deposition, desired composition

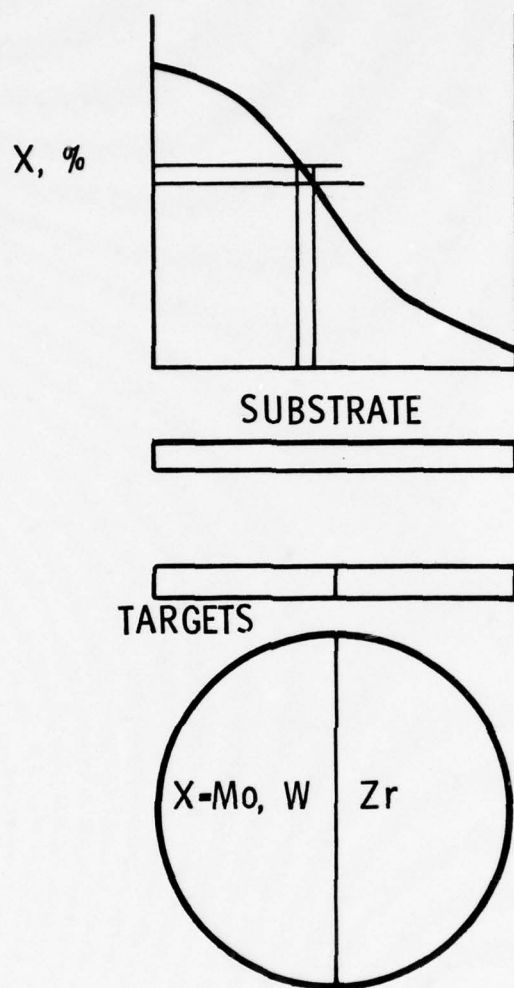


FIGURE 1. Sputtering target geometry used to produce deposits which would contain the composition  $X_2Zr$  ( $X = Mo, W$ ). The composition is shown schematically as a function of position on a diameter of the substrate.



the specimen was at  $1200^{\circ}\text{C}$  while the opposite end was at  $700^{\circ}\text{C}$ . After 20 minutes at temperature, a cluster of pale yellow whiskers was observed at the cold end of the tube. These were tentatively identified as  $\text{MoO}_3$  by x-ray diffraction. The weight loss of the  $\text{Mo}_2\text{Zr}$  specimen was less than observed in the open system experiments, presumably because of the limited oxygen available in the sealed tube. These results indicate that the anticipated  $\text{ZrO}_2$  surface film, which was expected to protect the compound from oxidation, is permeable to molybdenum. Upon diffusing to the surface, the molybdenum reacts to form the volatile oxide  $\text{MoO}_3$ .

#### $\text{W}_2\text{Zr}$

Due to the similarity of tungsten and molybdenum with respect to formation of volatile oxides, and the fact that the available tungsten-zirconium deposit was of poor quality, it was decided that testing the tungsten-zirconium system would not be in the best interest of the program.

#### $\text{Nb}_3\text{Al}$

A deposit of  $\text{Nb}_3\text{Al}$  was available from a superconductivity study in progress at this laboratory. Specimens about 1 cm square and 0.5 mm thick were cut from this deposit and vacuum annealed for 3 hours at  $750^{\circ}\text{C}$  to complete the compound formation reaction. One specimen was exposed to still air at  $1000^{\circ}\text{C}$  for 10 minutes. The specimen oxidized rapidly, converting about half its thickness to oxide in this time period. Lower exposure temperatures were investigated with

further specimens in an attempt to promote formation of the anticipated protective alumina film. The results were similar, i.e. no protective behavior was observed.

#### NbBe<sub>12</sub>

In view of the negative results obtained with the above materials, the material selection criteria were modified. The refractory metal beryllides,  $M_2Be_{17}$  and  $MBe_{12}$ , were known to have excellent oxidation resistance to temperatures over 2500°F.<sup>(6)</sup> Their phase diagrams, however, exhibit numerous lower beryllides, e.g.  $MBe_{2-6}$ , which suggests that a composite of the refractory metal M and the high beryllides may be unstable. The system Nb-NbBe<sub>12</sub> was selected for study of composite stability and oxidation behavior. Due to the high atomic fraction of beryllium in the desired compound, and the low sputtering yield of that metal relative to that of niobium, an asymmetric double "dee" target arrangement was used, Figure 2. The position of the division between the elements was selected on the basis of the yield data reported by Wehner.<sup>(7)</sup> Deposition conditions and results are listed in Table I. X-ray fluorescence analysis of the first deposit indicated that the desired composition occurred near the edge of the substrate, rather than in the central portion as intended. This resulted from an interaction between the low yield and high yield target elements, which had the effect of reducing the yield of the latter. A second deposit was made with a reduced target-substrate

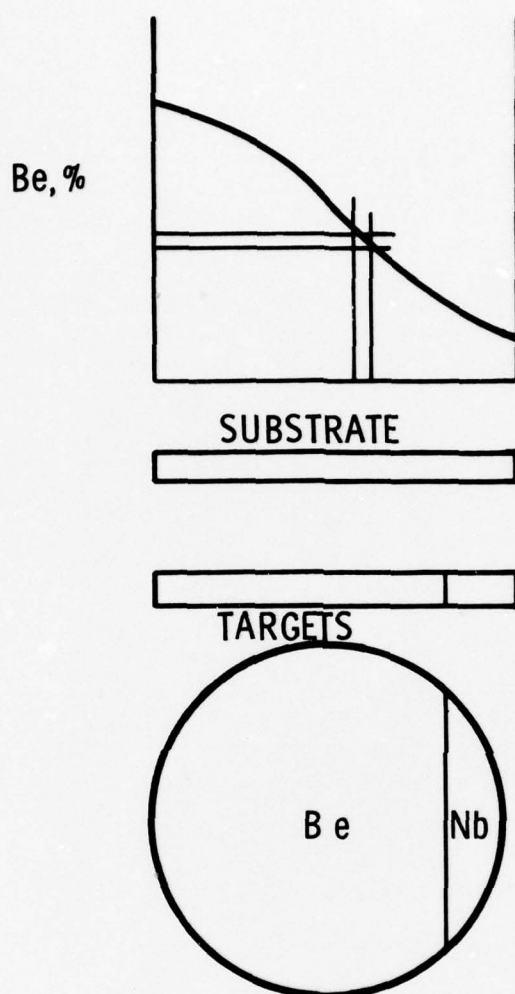


FIGURE 2. Sputtering target geometry used for deposits containing  $\text{NbBe}_{12}$ . The large relative yield ratio, together with the desired composition, required the asymmetric arrangement.

spacing, which resulted in shifting the zone of desired composition toward the center of the substrate.

Specimens were cut from the second deposit and removed from the substrate. They were vacuum annealed for 16 hours at 950°C to promote homogenization and completion of the compound forming reaction. X-ray diffraction indicated that only NbBe<sub>12</sub> was present. The annealed specimens were used for weight-gain experiments in still air at 1000°C. The results are shown in Figure 3. The time dependence is approximately  $t^{1/4}$ , although the first two points yield  $t^{1/2}$ , which is characteristic of the formation of a protective oxide film. The average rate of weight gain over the 100-hour period was 0.04 mg/cm<sup>2</sup> hr. The less than parabolic time dependence may be the result of vaporization of the sample at a low rate. Vaporization rates were not measured.

#### STABILITY OF Nb-NbBe<sub>12</sub>

An attempt was made to assess the stability of NbBe<sub>12</sub> in contact with Nb. A composite target was designed using the relative sputtering yields inferred from the deposits discussed above. A deposit of the NbBe<sub>12</sub> composition was formed on a niobium substrate. Sections of the coated substrate were examined by scanning electron microscopy and x-ray fluorescence after an anneal of 50 hours at 1200°C.

The structure of the annealed couple is illustrated in Figure 4 together with x-ray fluorescence analysis. A depleted region about 1.3 mils thick is observed in the



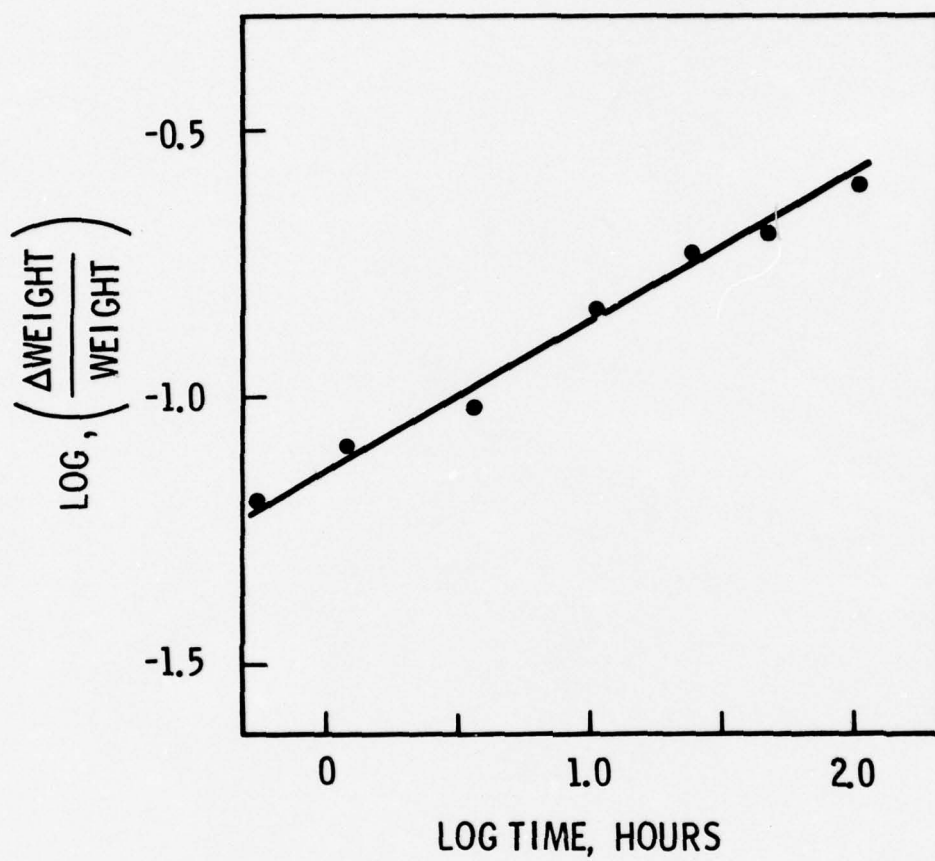
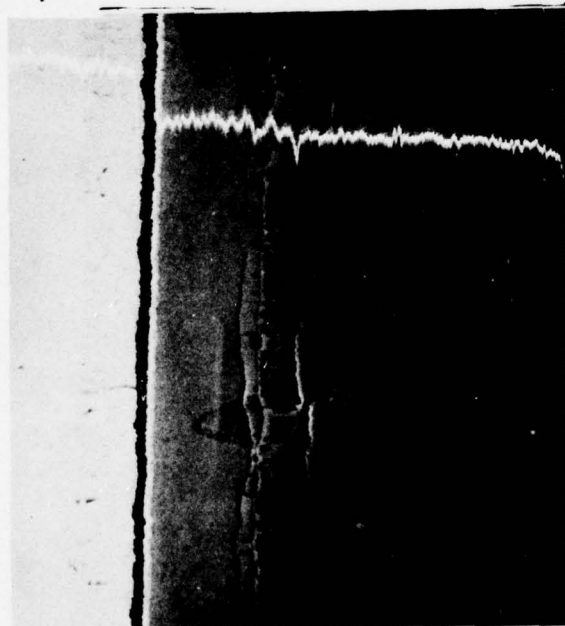


FIGURE 3. Oxidation of sputter-deposited NbBe<sub>12</sub> at 1000°C in 1 atm still air.

Line of scan  
↓



- 100 at.% Nb      Used as internal standard.
- 67.4 at.% Nb    Close to  $\text{Nb}_3\text{Be}_2$ .
- 15.2 at.% Nb    Continuous gradient.
- 14.1 at.% Nb    Continuous gradient.
- 12.2 at.% Nb    Continuous gradient.

↑  
125 μm

FIGURE 4. Nb-NbBe<sub>12</sub> diffusion couple annealed 50 hrs at 1200°C. SEM microstructure and x-ray fluorescence niobium trace. Compositions at several positions are indicated at right.

niobium substrate, and a 2.5 mil thick enriched (in niobium) band is observed in the deposit. An array of particles is present at the outer edge of the latter band. Although these particles appear enriched in niobium in Figure 4, averaging several scans at different positions showed that there was no consistent difference between the particles and the surrounding material.

The compositions of the layers were calculated using the intensity from a remote region of the substrate as a pure niobium standard. This procedure was checked by making a material balance between the substrate and deposit, from which the original composition of the deposit was determined to be 7.9 at.% Nb. This is in good agreement with  $\text{NbBe}_{12}$  (7.7 at.% Nb) as separately established by x-ray diffraction before the diffusion anneal. The derived compositions are shown at the right of the figure. With the exception of the depleted region of the substrate, which approximately corresponds to  $\text{Nb}_3\text{Be}_2$ , the experimental compositions do not correspond to the known compounds. This is taken as an indication that the diffusivity of niobium in the various compounds is roughly constant.

Kinetic data could not be obtained from the diffusion couple due to the interface cracking visible in Figure 4, which occurred during the anneal. However, the stability of the couple is obviously inadequate for use as a lamellar composite for high temperature service. Also, the cracking

parallel and perpendicular to the deposit-substrate surface would disqualify the material for load-bearing applications. The cracking is apparently due to stresses associated with specific volume changes during diffusion.

Tubular substrates were used for the deposition of lamellar composite specimens to be used for oxidation and stress-rupture testing. Due to the results of the deposition, which are described in the next section, the specimens obtained were not suitable for testing.

#### AIRFOIL FABRICATION DEMONSTRATION

##### OBJECTIVE

The objective of this portion of the program was to fabricate a small number of gas turbine blades consisting of a superalloy platform and root with a sputter-deposited lamellar composite airfoil. The airfoil material was to be selected from those investigated in the material development portion of the program. In the event that none of the candidate systems were successful, a eutectic system such as CoCrC,<sup>(8)</sup> or NiAl-NiNb,<sup>(9,10)</sup> which has been examined for turbine blade use in the directionally solidified condition, was to be used.

##### SPUTTER DEPOSITION APPARATUS

The sputter-deposition apparatus employed in the earlier phases of the program was modified to produce composite airfoils. The modification involved a larger chamber and a planetary gear system to rotate the specimens as they revolved



around the semicylindrical targets. The gearing was selected such that each specimen would make two complete revolutions while traversing each half of the chamber. Thus the composite would consist of a series of complete thin shells rather than a single continuous spiral. The apparatus was capable of coating up to twelve blades per loading, although only six positions were used in this work. The modified apparatus is shown in Figure 5.

#### SUBSTRATE FABRICATION

Substrates for the deposited airfoils were prepared using blades for a Pratt & Whitney FT7 gas turbine. The airfoil section was cut off about 0.5 cm above the platform and replicated in beryllium-copper by investment casting. The replica was vacuum brazed to the original root section with NIORO<sup>®</sup> foil and the new blade was hand-polished to ensure a smooth transition across the braze joint.

Deposition of the lamellar composite on these substrates was expected to bond the deposited material to the blade platform and the lower portion of the airfoil. After deposition, the beryllium-copper replica of the original airfoil was to be dissolved in acid. The sequence of operations is illustrated in Figure 6.

#### EXPERIMENTAL DEPOSITIONS

##### General

The turbine blade substrates and the deposition apparatus modifications were completed before the material development

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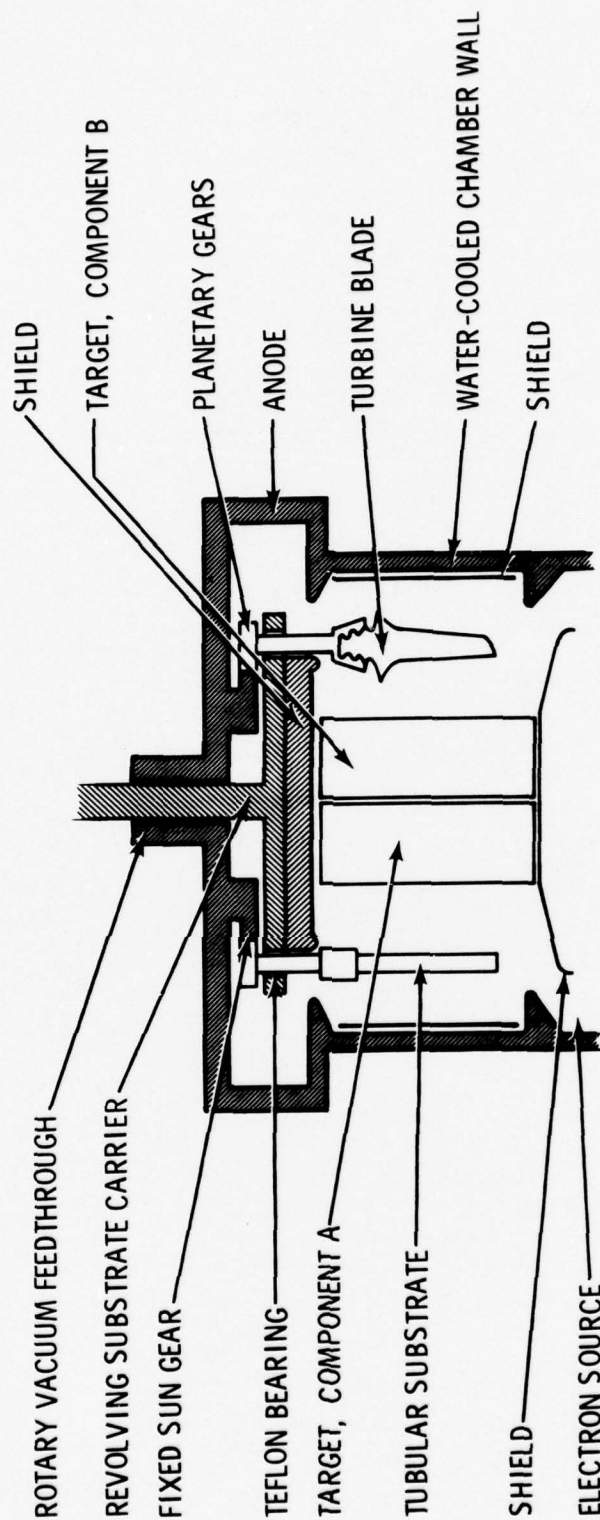


FIGURE 5. Arrangement of sputter-deposition apparatus for lamellar composite turbine blade and tubular test specimens.

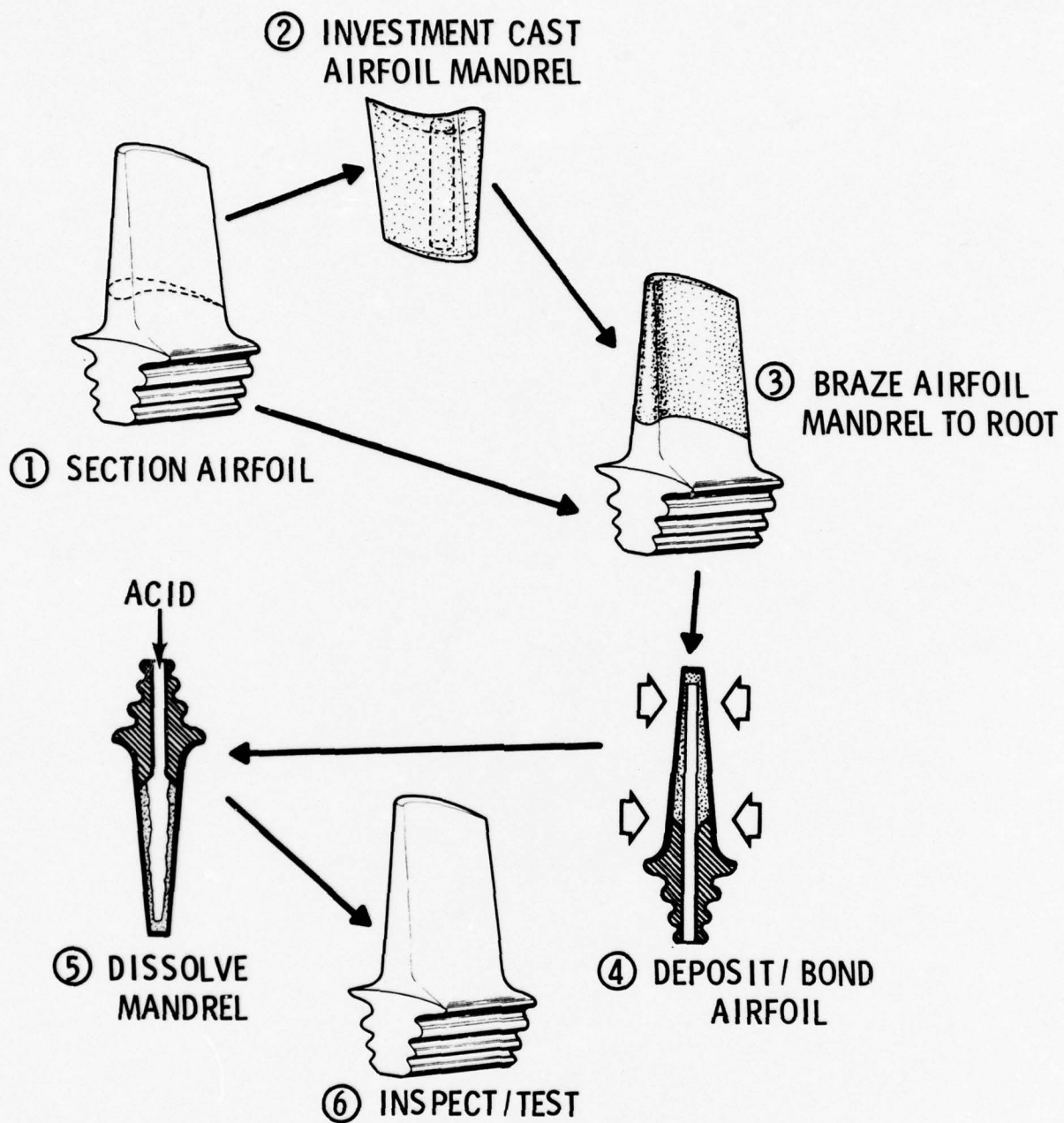


FIGURE 6. Turbine blade fabrication sequence.

work had reached the point of selecting a material system for blade fabrication. It was decided to use stand-in materials for the initial demonstration. Copper and molybdenum were selected since usable sputtering targets were available from earlier phases of the program. Several depositions were performed in an effort to overcome problems resulting from peeling of the stray deposit from shields in the apparatus.

During this period, the Nb-NbBe<sub>12</sub> system had been selected for blade fabrication, and sputtering targets were procured. When these became available they were substituted for the copper and molybdenum stand-ins. In subsequent depositions, a mixture of turbine blades and tubular specimens were coated; the latter to be used for oxidation and stress rupture testing. These depositions also suffered shield peeling problems resulting in premature terminations.

Finally, as program funds were nearly expended, depositions were made to at least demonstrate the fabrication of airfoils of a single material. Stainless steel was selected primarily on the basis of compatibility with acid solution of the copper mandrel.

These depositions are summarized in Table II.

#### Copper-Molybdenum Depositions

In the first deposition, No. 26, Table II, a preliminary layer of titanium was deposited on the anodes and the principal shields in the sputtering chamber. This technique had solved the peeling problem from these surfaces in earlier work on this program. It did not, however, prevent peeling



TABLE II. Depositions for Demonstration of Composite Airfoil Fabrication

Exp. No.	Target Materials	Target Amp-Hrs	Grams Deposited on Airfoil	Special Procedures	Comments
26	Cu-Mo	7	1.5	Deposited titanium on shields	Shield peeling adjacent to Mo target
27	Cu-Mo	23	4.7	Ion etch shields	Ditto
28	Cu-Mo	8	1.7	Ion etch shields, intensity	Ditto
29	Cu-SST	7	1.7	Ion etch shields as for 28	Target shield peel- ing, adverse effect of shield etching discovered
30	Nb-NbBe	41	1.5*	Test of several shield materials and temperatures	Hot shields of nickel or copper showed no peeling
31	Nb-NbBe	81	5.6	Nickel foil shields	Deposit debonded apparently due to high internal stresses
32	Nb-NbBe	18	0.7	Attempt to reduce stress in deposit	As for 31
33	SST	40	6.6	Nickel foil shields	Interrupted by com- puter malfunction; shield peeling when resumed
34	SST	37	6.1	Nickel foil shields, substrate -30 V	Interrupted by line transients; shield peeling when re- sumed

\*Tubular substrates for materials experiments.

of the stray molybdenum deposit in the present case. The deposit on the airfoils, which were ion etched prior to deposition, and which were not coated with titanium, was adherent.

To prevent peeling, deposition No. 27 was begun by ion etching all possible surfaces (i.e. those not permanently grounded by their construction) in the sputtering chamber. Both anodes and the principal shields were sequentially etched. Then the substrates (airfoils) were etched and deposition commenced. The run was terminated by shorting of the target after 5 hours. The portion of the shields adjacent to the molybdenum target had peeled in small flakes, some of which were responsible for the target short.

Deposition No. 28 utilized the same technique, with increased ion etching of the chamber shields. The stray molybdenum deposit again peeled, shorting the target and terminating the run.

#### Copper-Stainless Steel Depositions

The above experience prompted a substitution for the molybdenum. Stainless steel was selected on the basis of immediate availability and compatibility with the eventual acid solution of the copper mandrel. Deposition No. 29 again utilized the ion etching of the chamber anodes and shields. It was terminated by shorting of the electron source. Examination of the stray deposit causing the short circuit indicated that the multiple step etching of chamber

surfaces was responsible for the lack of adherence in this instance. The etching produced thin layers of the shield and substrate materials on all surfaces of the chamber. In areas where these layers were deposited at high angles to the surface normal, the deposit structure was porous and poorly bonded, resulting in low strength. When further material was deposited over this weak layer during normal sputtering, peeling resulted. The multiple step ion etching was therefore abandoned in subsequent depositions.

#### Nb-NbBe Depositions

Deposition No. 30 was primarily an investigation of the influence of shield material and temperature on the adherence of the stray deposit. Strips of candidate materials were spot welded in place in the chamber in a manner which ensured they would reach elevated temperatures during deposition. Stainless steel mesh (~ 3 mm) was also employed as an auxiliary shield in the expectation of limiting the effects of peeling from adjacent surfaces.

Niobium/niobium-beryllium targets were employed in this deposition so that if one or more candidate shield materials proved successful, the result would be directly applicable for the remainder of the program. Copper tubes were used as substrates to provide material for metallographic examination and testing.

The deposition ran for 15 hours before a short circuit developed. Peeling occurred from the tantalum and stainless

steel strips in areas adjacent to the niobium target, and particularly the area adjacent to the target dividing line. The stray deposit on the nickel and copper strips, the cold copper shield exposed between strips, and the stainless steel mesh, was adherent. The nonadherent deposit was copper colored on the debonded surface. This indicates that the material removed from the substrates during the ion etch comprises a weak interface, at least in the case of tantalum and stainless steel surfaces.

The deposit on the copper substrates was initially adherent, but began to crack after the apparatus was opened. The cracking was followed by peeling, but the occurrence was interpreted as being due to very high stresses in the lamellar material rather than poor bonding to the substrate. Three of the six substrates were affected, suggesting that whether the initial material deposited was niobium or niobium-beryllium was significant in determining the bond strength.

Deposition No. 31 employed shields fabricated from nickel sheet, placed in the apparatus in a manner such that they would run at an elevated temperature. Three airfoils and two copper tubes were used as substrates; the sixth substrate position was used for a nonrotating thermocouple housing. The deposition continued for twenty-one hours before being terminated by a massive arc which destroyed power supply components. Although peeling was observed when the apparatus was opened, it could not be determined whether this peeling caused the termination or occurred after the



termination upon cooling of the apparatus. The deposits cracked and peeled extensively on the airfoils and to a lesser extent on the copper tube substrates. As in the previous deposition, this cracking/peeling was associated with high internal stress in the deposits.

In an effort to reduce these stresses, for deposition No. 32, the voltage applied to the Nb-Be target was reduced by 40% to similarly decrease the amount of the reinforcing phase. The planetary rotation was slowed from 1 rpm to 0.2 rpm, to increase the thickness of individual niobium layers. Also, the substrates were electrically connected through a variable resistor to the anode, thus permitting controllable temperature increases due to electron bombardment. Finally, the Nb-Be target was used singly for the first 20 minutes of deposition to ensure a common initial layer on all substrates.

The described methods apparently did not reduce the stress level, since the deposition was terminated by peeling in only five hours. The deposit on the airfoil substrates peeled off in large flakes, while the stray deposit on the shields was adherent except for the areas adjacent to the target dividing line.

#### Stainless Steel Depositions

During the course of the above experiments, investigation of the thermal stability of the Nb-NbBe system was completed. Those negative results, together with the difficulties due

to deposit stress and shield peeling discussed above, prompted the adoption of the less ambitious goal of demonstrating the fabrication of the airfoil with a single material rather than a lamellar composite. Stainless steel was selected on the grounds of compatibility with the soluble mandrel scheme and availability of a sputtering target.

Two depositions of stainless steel were performed to build up the thickness of the airfoil section to a level which would permit handling after dissolution of the copper mandrel. Although no peeling problems were anticipated with the use of a single target material, the nickel foil shield system, which had been found effective in minimizing such problems, was used in these depositions.

The first deposition, No. 33, ran normally for about six hours, at which time a computer malfunction reduced the system power to a negligible value for the remainder of the offshift period. The power was reset at the beginning of the next day shift, and the run continued for an additional seven hours before being terminated by shorting. It appeared from the thickness of the peeling material that the long period of operation at very low power produced a plane of weakness in the material deposited on the shields.

Deposition No. 34 was also prematurely terminated by short circuiting from flakes of debonded shield deposit. On two occasions during the deposition, the process control computer had reduced system power to zero. This behavior

was traced to the effect of voltage transients in the laboratory electrical system, which triggered the computer's power failure response. The deposition remained shut down until manual control was assumed, whereupon deposition proceeded onto surfaces which had been exposed to a low vacuum environment for periods of a few minutes to ~ ten hours. The resulting deposit was only weakly bonded, and peeled when a moderate thickness was attained. Peeling did not occur on the substrates, which were ion etched before resuming deposition.

These depositions resulted in a total of about 15 mils airfoil thickness, which was considered sufficient for demonstration purposes. The copper mandrels were dissolved in nitric acid which left the composite airfoil bonded to the superalloy platform and root section. One of the completed blades is illustrated in Figure 7.

Although the shield peeling and deposit stress problems discussed above prevented the use of lamellar composites, airfoil fabrication and bonding to a root section was demonstrated. The bond strength was not quantitatively evaluated, e.g. by spin testing, since the property would be expected to depend on the deposit character, and therefore the data obtained would not be relevant to a composite system.

#### FUTURE WORK

It appears that the best way to establish the utility of this airfoil fabrication process would be to use an established composite material system such as an eutectic

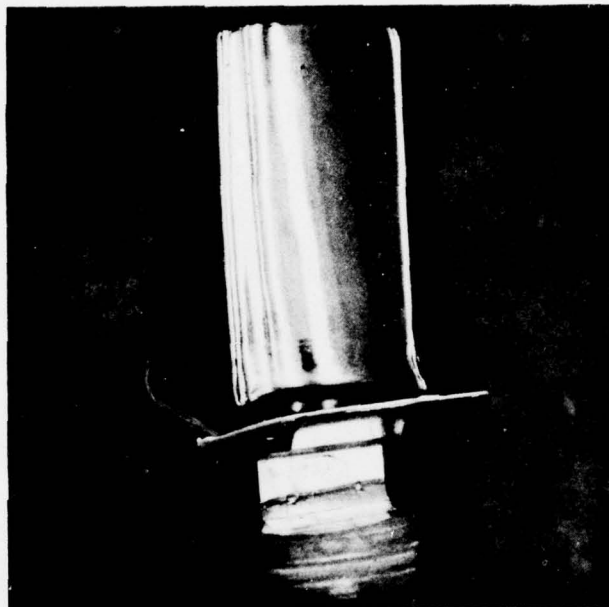


FIGURE 7. Turbine blade with sputter-deposited airfoil bonded to cast platform-root section by the soluble mandrel method. 1X



system normally fabricated by directional solidification.

The deposition method has the following advantages:

- 1) The composition may be altered to either side of the eutectic point, thus permitting control of the volume fraction of the reinforcing phase and the mechanical properties. If desired, this variation can be accomplished across the airfoil wall thickness, producing, for example, a more ductile material adjacent to the outside surface.
- 2) The layer thicknesses of the composite structure are subject to direct control independent of the rate of the fabrication process.
- 3) Protective coatings could be applied on both the inner and outer surfaces as part of the fabrication process. Also, the composite structure could be arranged to minimize reaction between coating and blade, as has been observed in systems containing carbides. (11)

An additional characteristic involves the "sheet" nature of the deposited composite system as compared to the rod or lath nature of the composite structure formed by directional solidification. At the present time it is not clear that this characteristic would be favorable. The expected equality of the longitudinal and transverse mechanical properties may not be significant for the turbine blade application, with its predominantly uniaxial loading. Also,

the presence of continuous layers of the reinforcing phase may be deleterious to the impact and fatigue properties.

This question should be resolved in future work, together with quantitative measurement of the airfoil/root section bond strength for the composite system.

#### SUMMARY AND CONCLUSIONS

The objectives of this phase of the lamellar composite program were two -- to investigate lamellar systems based on a refractory metal and an intermetallic compound of the refractory metal and a strong oxide-former, and to demonstrate the fabrication of a thin wall turbine blade from such a lamellar composite by sputter deposition.

Three material systems were initially selected for examination, based upon average melting point and the degree of thermal stability of the system anticipated on the basis of the phase diagram. The systems were W-W<sub>2</sub>Zr, Mo-Mo<sub>2</sub>Zr and Nb-Nb<sub>3</sub>Al. The expected formation of a protective oxide layer, e.g. ZrO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, did not occur. In Mo<sub>2</sub>Zr, the Mo was found to be completely consumed through volatilization of MoO<sub>3</sub>; similar behavior was thus expected for W<sub>2</sub>Zr. The compound Nb<sub>3</sub>Al oxidized rapidly at temperatures over 700°C, forming a solid complex oxide.

A fourth system was therefore selected, based on NbBe<sub>12</sub>, which was known to exhibit very low oxidation rates up to 1400°C. The composite system, Nb-NbBe<sub>12</sub>, was found to be unstable to diffusion processes at 1200°C, as would be expected from the phase diagram.

The demonstration of thin wall turbine blade fabrication by sputter deposition was carried out with substitute materials. Initially, copper and molybdenum were selected on the basis of availability of sputtering targets, and compatibility with the soluble mandrel fabrication scheme. Severe problems were experienced with nonadherent deposits on various shield surfaces in the sputtering apparatus. When sputtering targets for the niobium-beryllium system became available, the fabrication was continued with this system. The internal stresses in the lamellar composite deposits were sufficient to cause cracking and debonding from the airfoil substrates. Attempts to reduce this stress by reducing the volume fraction of the reinforcing phase, increasing the substrate temperature, etc. were not sufficiently effective to prevent the cracking.

Finally, the lesser objective of demonstrating the blade fabrication scheme with a single material rather than a lamellar composite was adopted. The airfoil deposits were completed to sufficient thickness for handling purposes with stainless steel, and the mandrels were dissolved out, leaving the sputter deposited wall bonded to the superalloy platform and root section.

The basic soluble mandrel scheme was successfully demonstrated. However, because of the difficulties described above, the intended deposition format was not achieved, and thus the critical area of the bond strength between the deposited airfoil and the root section was not quantitatively

examined. This area cannot practically be separated from the selection of the airfoil (composite) material, as illustrated in the case of the Nb-NbBe deposits, which debonded as a result of their high internal stress levels.

The logical continuation of this work would utilize the soluble mandrel fabrication scheme with an existing composite material system, such as one of the eutectic systems normally produced in composite form by directional solidification. This would permit detailed evaluation of the bonding and mechanical characteristics of thin wall lamellar composite airfoils.

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